Parallel Reactor Standard Operating Procedure

THIS SOP IS NOT MEANT TO REPLACE HANDS-ON TRAINING FROM QUALIFIED PERSONNEL!

This SOP describes the basic procedure for using the HEL Cat-24 parallel reactor, routine maintenance, common problems, and their solutions.

General Comments

Treat the pressure reactors with respect at all times. If you are at all unsure of something, ask for help. NEVER WORK ON A PRESSURIZED SYSTEM!

The volume of each well in the reactor base is 2 mL. Maximum reaction volume is 1.5 mL. Disposable 2 mL borosilicate glass tubes line each well. Generally, 20 reaction samples and 4 blanks comprise the reaction array.

The reactor has a burst disc rated to approximately 1750 psi at room temperature. Maximum working pressure should be no more than 1400 psi. The usual operating pressure for the parallel reactor is 1100 psi at room temperature. This translates to about 1400 psi at 200 °C.

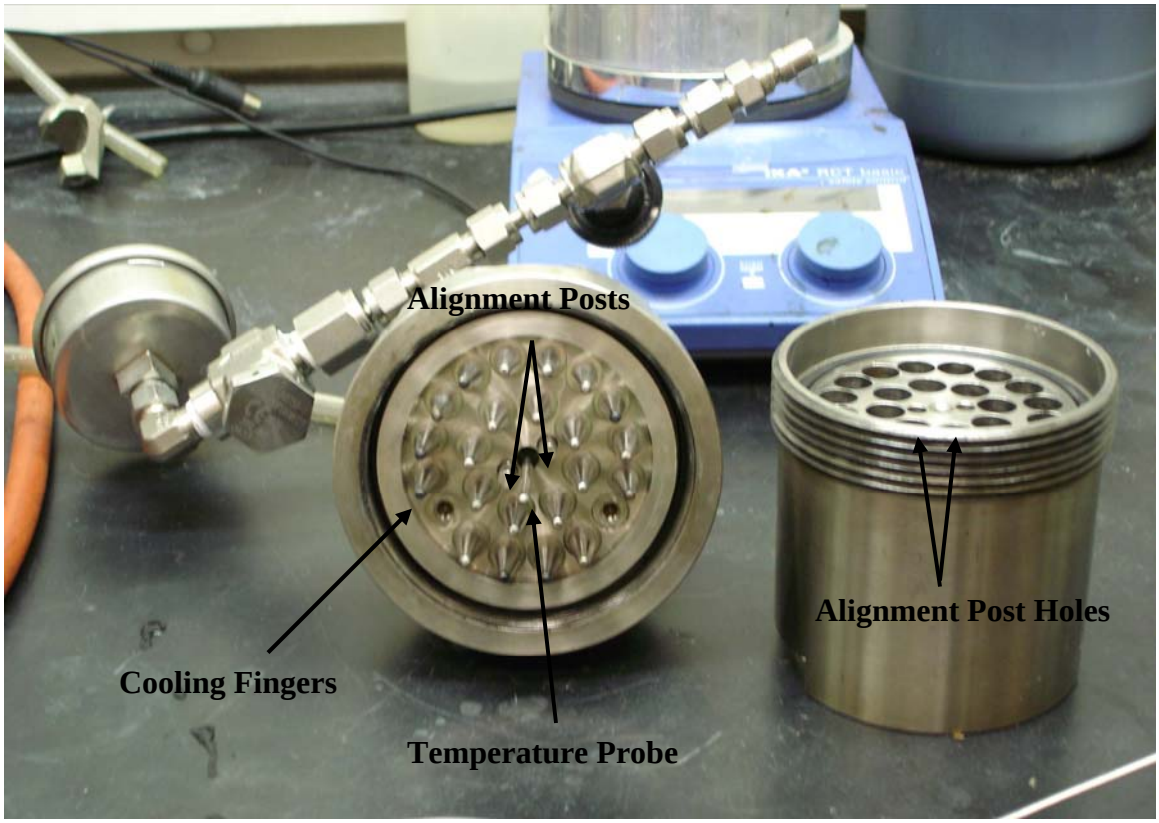
Hydrogenation experiments are ONLY performed under STATIC pressure. NEVER do a reaction under dynamic hydrogen flow. The lab is not equipped with the proper controls and safety features for such experiments.

The parallel reactor is equipped with its own hydrogen tank, generally regulated to 1100 psi. When not in use, the reactor is disconnected from the tank, the main tank valve is closed, and the pressure is bled from the regulator.

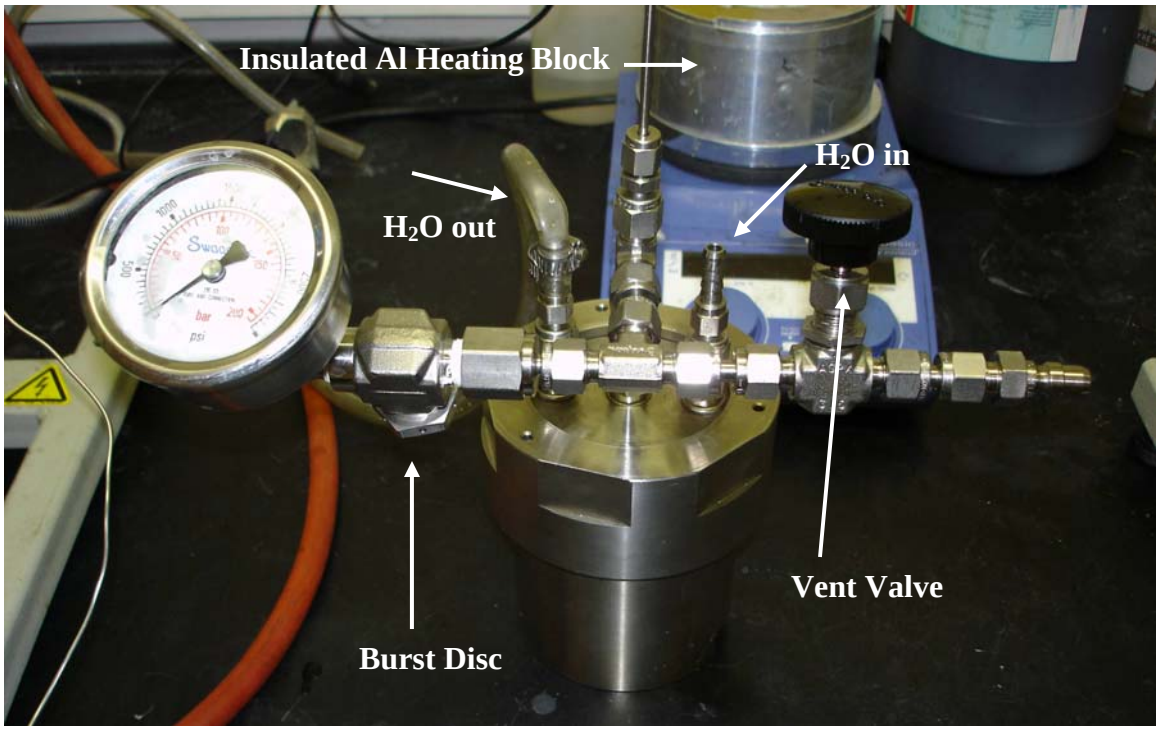
Familiarize yourself with the Pressure Reactor Checklist, posted in the hydrogenation lab.

The effective temperature limit of the parallel reactor is 200 °C with the cooling water running. A glass wool-insulated aluminum heating block is used to achieve and maintain this temperature. Without the cooling water running, the empty reactor can reach at least 250 °C and hold pressure overnight. Cooling water is necessary to prevent movement of the more volatile products between wells.

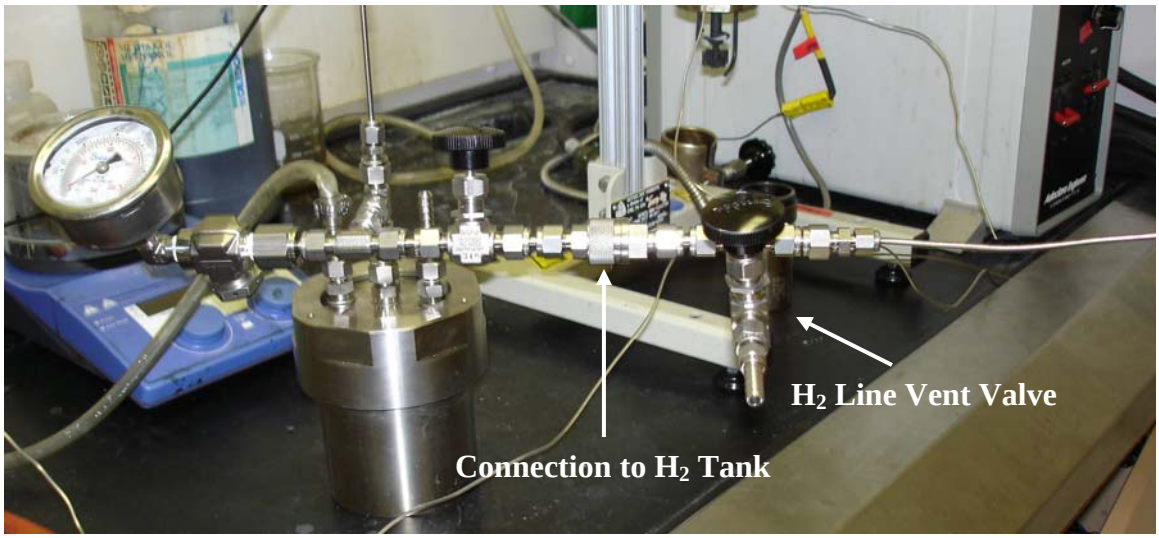
The Parallel Reactor



Parallel Reactor



Parallel Reactor, Closed as During Operation



Parallel Reactor, Connected to the H_2 Tank for Pressurization

Setup

1. Prepare the reaction array, including the blanks, in 2 mL disposable test tubes. The tubes are not labelled. A labelled foam block with multiple holes or test tube rack is used to keep the samples in order. Add a 2 × 2 mm stir bar to each test tube. To ensure complete homogeneity, mix each tube thoroughly using a vortex mixer. Take a 0.5 mL sample for initial GC analysis from all but the blank samples.

2. Load the tubes into the parallel reactor. The letter "A" is scratched by one of the wells in the outer circle as an arbitrary starting point. The letter "Q" is scratched by one of the wells in the inner circle. For consistency and to avoid confusion, it is best to always load the tubes in the same direction (clockwise or counter-clockwise). Close the reactor. The alignment posts must fit into the alignment post holes. The inner part of the reactor top will sit flush on the reactor base. Clamp the base in the vise and tighten the outer screw ring by hand. The reactor can generally be sealed by hand strength alone, but if necessary the boa wrench can be used. Remove the reactor from the vise and place it in the fumehood.

3. Connect the H_2 line to the reactor. Pull the knurled nut back, slide the line onto the reactor vent, and release the nut. Attach the aspirator hose to the H_2 line vent valve. Evacuate the reactor for 2 minutes using the water aspirator, then close all the valves, disconnect the aspirator hose, and turn off the water. Turning the water off before removing the hose can result in water being sucked into the reactor!

4. Pressurize to 1100 psi with H_2 gas, and let equilibrate for 2 minutes. Close the needle valve on the H_2 tank after pressurizing the reactor. To vent, first open the vent valve on the H_2 line, then slowly open the vent valve on the reactor. Leaks on the parallel reactor are rare, and generally only occur if it is not completely sealed. If that is the case, close the main valve on the hydrogen tank, vent the reactor, and disconnect it

from the hydrogen line. Tighten the screw ring. Pressurize and check for leaks again. NEVER WORK ON A PRESSURIZED SYSTEM!

5. Repeat the evacuation/pressurization cycle twice more. After the final pressurization, close the needle valve on the tank as usual, but vent the H_2 line only. Disconnect the H_2 line from the reactor by pulling back the knurled nut to release the line.

6. Place the reactor in the glass wool-lined aluminum heating block on the hotplate, begin a gentle flow of cooling water (about ¼ turn of the tap), turn magnetic stirring on to the maximum, set the reaction temperature, and begin heating. Wait until the set temperature has been reached.

7. BEFORE LEAVING THE HYDROGENATION LAB, ensure the main valve is closed on the hydrogen tank, the regulator is vented, the fumehood sash is lowered, and the safety chains are hooked.

8. At the end of the reaction, turn the heat off and place the reactor in an ice bath for 30 min, followed by a dry ice/acetone bath for 5 min to condense and freeze any volatile products. Vent the reactor and warm to room temperature before opening. Transfer the remainder of the solutions in the test tubes to GC vials for analysis.

9. Clean the parallel reactor by rinsing the base of the reactor and the cooling fingers with methanol. Discard the glass test tubes, and rinse the stir bars several times with methanol. Periodically stir the stir bars in a few mL of *aqua regia* to remove deposits that may have accumulated. Leave the reactor assembly open to air dry overnight. Turn the hotplate off. MAKE SURE THE HYDROGEN TANK IS CLOSED AND THE REGULATOR BLED OUT.

Routine Maintenance

1. Regrease the threads on the reactor base and the inner ring of the reactor top every 10 experiments or as needed using the Moly grease (molybdenum disulfide). A little goes a long away! More grease can be obtained from the machine shop.

2. The cooling fingers tend to loosen over time. Periodically check the reactor top and tighten any loose fingers.

3. Excessive black deposits can be removed from the cooling fingers by unscrewing them and sanding them with fine sandpaper.

4. Inspect the Viton O-ring periodically for wear. A new one can be obtained from the machine shop.

Problems & Their Solutions

LEAKS: Use Snoop to find. Close the main valve on the hydrogen tank. Vent the reactor. Tighten the fitting(s). Pressurize and check for leaks again. NEVER WORK ON A PRESSURIZED SYSTEM!

BURST DISC: Have the machine shop replace the burst disc if it blows. They will supply, or can order, a replacement burst disc from Swagelok.